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JAN 79 S B WARNER, L H PEEBLES, D R UHLMANN N00014-75-C-0542

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Stabilized acrylic fibers are very hygroscopic. Dried fibers rapidly equilibrate under ambient condition with a gain in moisture of about 8 wt. percent and concurrently elongate about 1.5 percent.

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OXIDATIVE STABILIZATION OF ACRYLIC FIBERS
IV. MOISTURE SENSITIVITY

by

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OXIDATIVE STABILIZATION OF ACRYLIC FIBERS

IV. MOISTURE SENSITIVITY

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ABSTRACT

Stabilized acrylic fibers are very hygroscopic. Dried fibers rapidly equilibrate under ambient conditions with a gain in moisture of about 8 wt pct. and concurrently elongate about 1.5%.

A number of accounts in the literature on producing carbon fibers allude to the moisture sensitivity of stabilized acrylic fibers (see, e.g., Refs. 1 and 2). Indeed, Kinoshita (3), uses the moisture regain at 25°C, 81% RH as a measure of the extent of stabilization: fibers with less than 5% regain are understabilized, those with more than 15% regain are over-stabilized. But to date no detailed account of moisture regain has been presented. This paper reports such data and directs attention to some of the important consequences of the pronounced moisture sensitivity of these materials.

The equilibrium moisture regains (under ambient conditions) of four acrylic fibers heat treated in air at 255°C for various times while wrapped about a glass mandrel are shown in Fig. 1. Virgin acrylic fibers are seen to absorb 0.5-1.0 pct. by weight of water, in agreement with previously published data. Well-stabilized fibers absorb 8 pct or more by weight. The most remarkable features about Fig. 1 is the similarity in the shape of these curves to those of oxygen uptake (4, 5).

The dynamic moisture regain of well-stabilized Monsanto acrylic and Courtelle is shown in Fig. 2 (c.f. Ref. 4 for details on fibers). The data points were determined as follows: (1) Fibers were heat treated at 255°C

in air while wrapped about a glass mandrel; (2) after 230 minutes of heat treatment time, the fibers were removed from the furnace and separated from the mandrel;(3) the fibers were reheated to about 150°C for several hours, a treatment which has been found to eliminate all but tenaciously bound water; and (4) the fibers were removed from the drying oven and immediately placed on a Mettler balance where weight was monitored as a function of elapsed time.

The data indicate that the uptake of moisture occurs with similar kinetics in stabilized material, independent of the precursor. Within just a few minutes of exposure to the ambient (in excess of 50 pct. relative humidity), the fibers absorb a considerable amount of water. This behavior can be of major importance, for example, when analyzing the fibers for oxygen.

Fig. 3 shows the effect of moisture content on the measured length of the stabilized fiber. The designation "Gas ON" indicates the time when the relative humidity of the environment around the fibers was changed from ambient to essentially zero by flowing dry forming gas (5 pct. hydrogen; the balance, nitrogen) around the fiber. The designation "Gas OFF" indicates the time when the gas was shut off and ambient air was allowed to diffuse back into the vicinity of the fiber tow. The length changes were measured directly with a cathetometer; the fibers were subjected to a stress of 5-12 x 10^{-3} gms denier^{-1} (0.5-1.2 MPa) during the experiment.

Clearly, the uptake of moisture is accompanied by expansion of the fibers, as is the case with cotton, wool and nylon even at temperatures as high as 160°C. The data indicate the occurrence of another phenomenon which

may have important consequences in processing. As the temperature of a stabilized fiber is cycled about 90-100°C, the length of the fiber changes dramatically. Hence when a fiber is cooled from the reaction temperature, it expands in length. On a molecular level, the process may be envisioned as follows: Polar groups which bind molecular segments in the polymer are solvated by the water, and Van der Waals bonds are formed between the polymer and the water. In this way the polymer is allowed to relax or swell, leading to fiber extension. Any model of the stabilized material must, therefore, have extensive secondary bonding.

In summary, the strong secondary forces between molecular segments in stabilized acrylic fibers make the material very hygroscopic. Upon exposure to moisture the stabilized acrylic fibers rapidly equilibrate with a gain in moisture of about 8 wt. pct. and concurrently elongate about 1.5 pct. As they exist in the laboratory, the stabilized fibers can therefore be regarded as a plasticized material.

ACKNOWLEDGEMENTS

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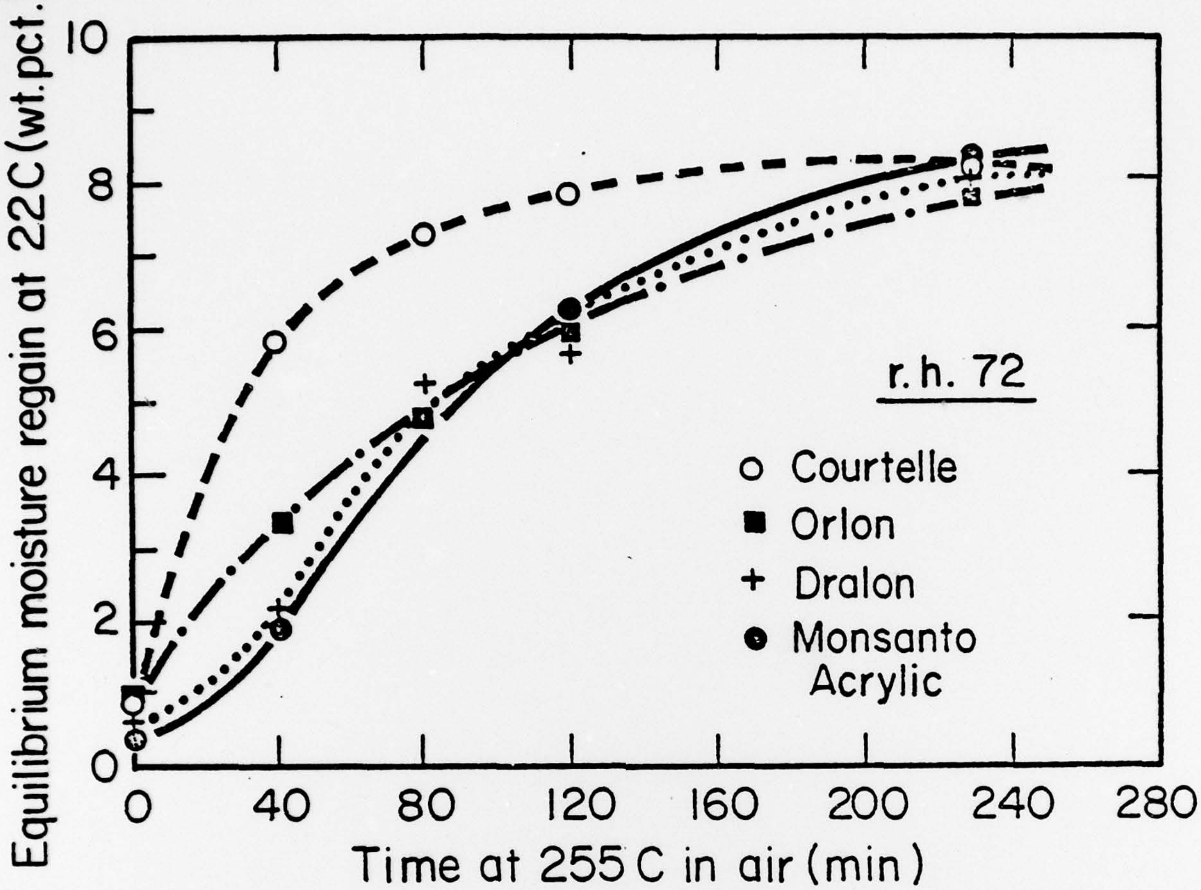
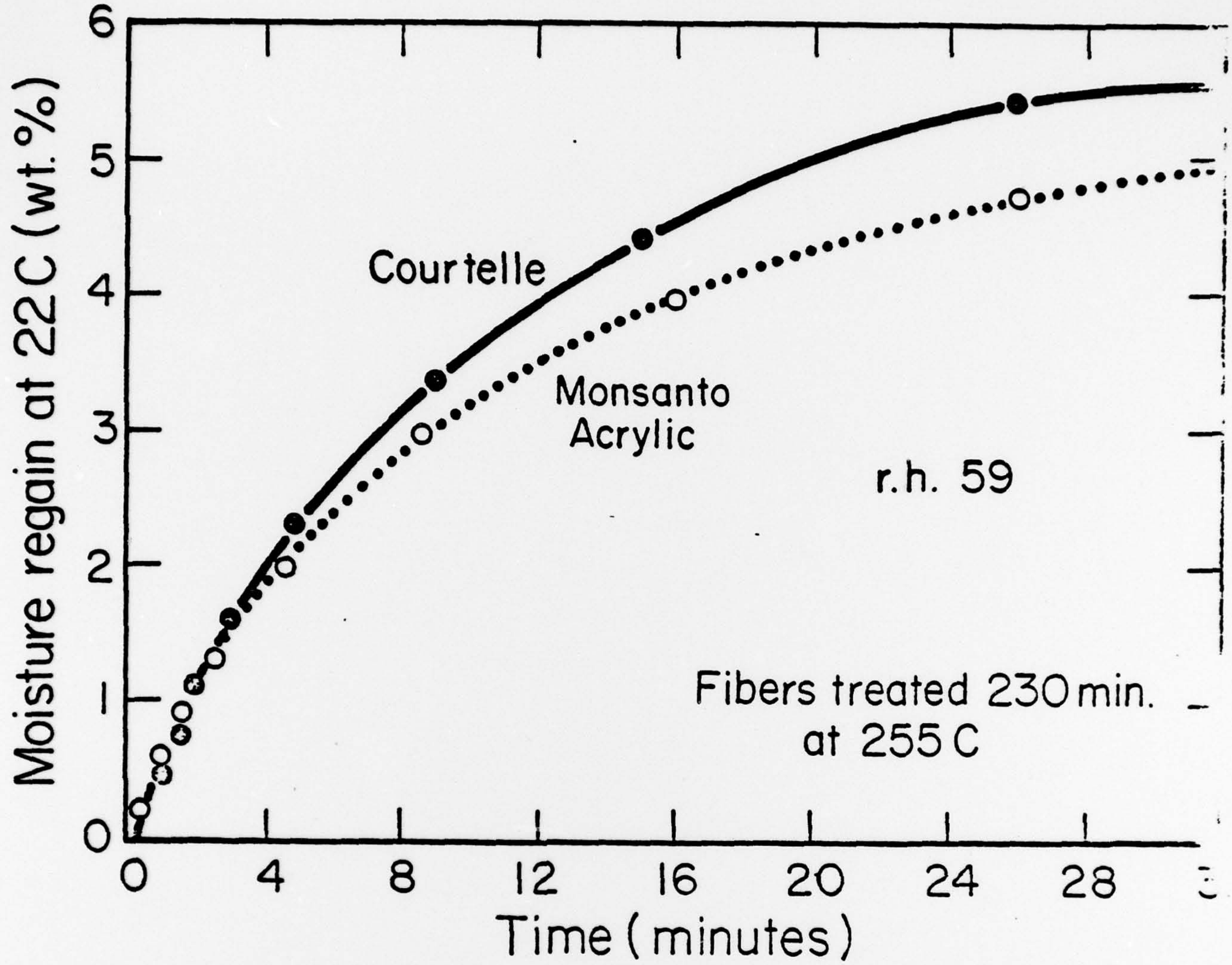
FIGURE CAPTIONS

Fig. 1 Equilibrium moisture regain at 22C (wt. pct.)

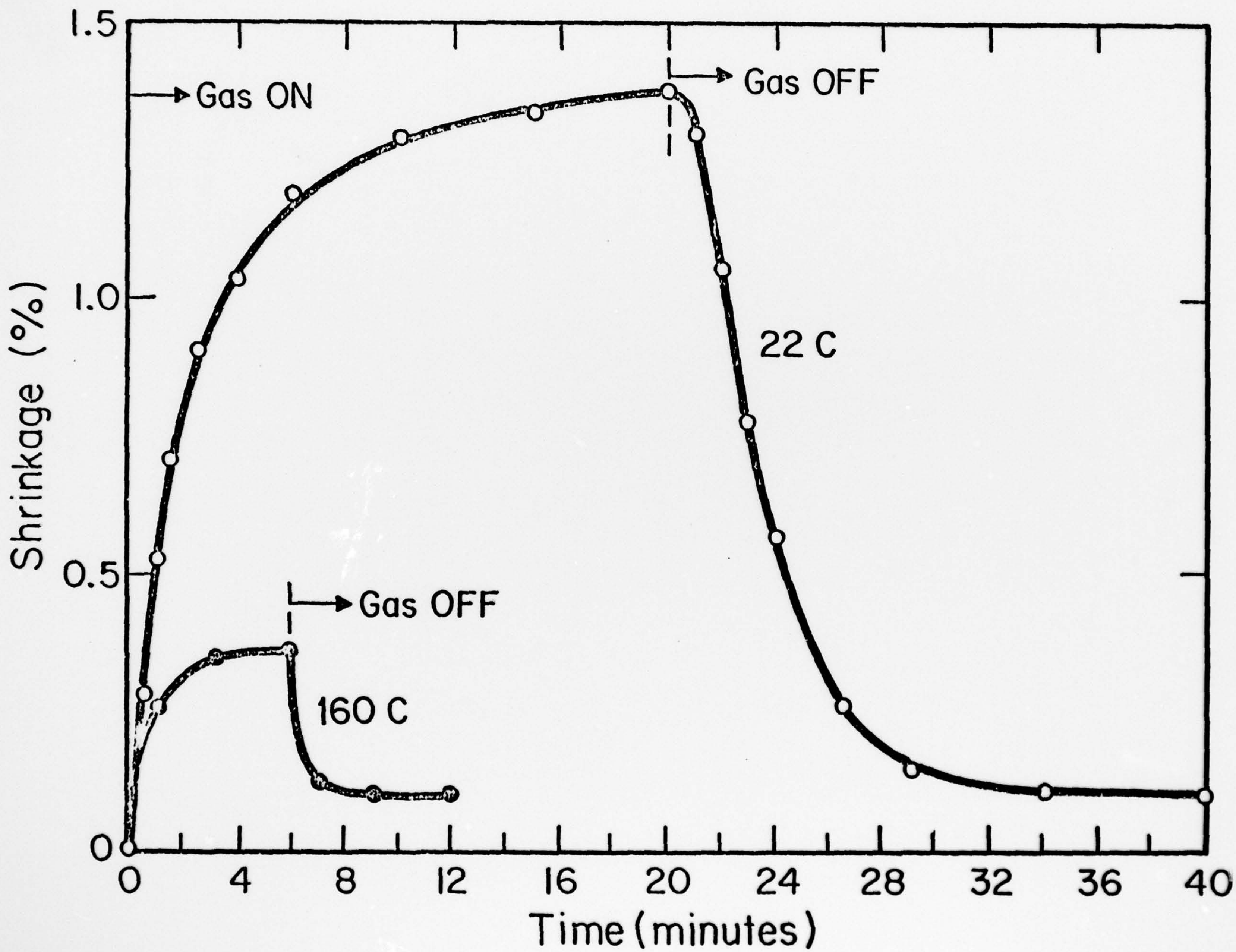
Fig. 2 Moisture regain at 22C (wt. %)

Fig. 3 Effect of relative humidity on isothermal length of stabilized Monsanto acrylic at 22° and 160°


Equilibrium moisture regain at 22C (wt.pct.)
Time at 255 C in air (min)
0
2
4
6
8
10
0
40
80
120
160
200
240
280
r.h. 72
Courtelle
Orlon
Dralon
Monsanto Acrylic



Moisture regain at 22 C (wt. %)
Time (minutes)
Courtelle
Monsanto Acrylic
r.h. 59
Fibers treated 230 min. at 255 C
0
1
2
3
4
5
6
0
4
8
12
16
20
24
28



Gas ON
Gas OFF
22 C
Gas OFF
160 C
Shrinkage (%)
Time (minutes)
0
0.5
1.0
1.5
0
4
8
12
16
20
24
28
32
36
40